

# Structural and shielding properties of NiO/xCo<sub>3</sub>O<sub>4</sub> nanocomposites synthesized by microwave irradiation method

Atif Mossad Ali<sup>a</sup>, Shams A.M. Issa<sup>b,c</sup>, Hesham M.H. Zakaly<sup>c,d</sup>, Mariia Pyshkina<sup>d,e</sup>, H. H. Somaily<sup>a</sup>, H. Algarni<sup>a</sup>, M. Rashad<sup>b,f</sup>, M. Saif<sup>d</sup>, H.A.A. Sidek<sup>g</sup>, K.A. Matori<sup>g</sup>, M.H.M. Zaid<sup>g,\*</sup>

<sup>a</sup> Physics Department, Faculty of Science, King Khalid University, Abha, Saudi Arabia

<sup>b</sup> Department of Physics and Nanotechnology Research Unit, Faculty of Science, University of Tabuk, Tabuk, Saudi Arabia

<sup>c</sup> Physics Department, Faculty of Science, Al-Azhar University, Assiut 71524, Egypt

<sup>d</sup> Institute of Physics and Technology, Ural Federal University, Yekaterinburg, Russia

<sup>e</sup> Institute of Industrial Ecology UB RAS, Ekaterinburg, Russia

<sup>f</sup> Physics Department, Faculty of Science, Assiut University, Assiut 71516, Egypt

<sup>g</sup> Department of Physics, Universiti Putra Malaysia, 43400 Serdang, Selangor, Malaysia

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## ABSTRACT

In the present study, nanocomposites with different ratios of NiO and Co<sub>3</sub>O<sub>4</sub> ( $x = 0.3, 0.5$ , and  $0.7$ ) have been prepared via microwave oven and characterized using XRD. FLUKA code has been used to estimate the values of the mass attenuation coefficient ( $\mu_m$ ) for all samples. From the measurement, we found that when the thickness of the samples increases, the gamma transmission values decrease. Besides, the  $\mu_m$  values increase as the Co<sub>3</sub>O<sub>4</sub> content increase from 0.3 to 0.7%. At selected photon energy, the HVL, TVL, and MFP values decrease with increasing Co<sub>3</sub>O<sub>4</sub> concentrations. At low energies (0.1–0.5 MeV), the linear decreasing trend in MAC values indication that photoelectric effect (PEA) ( $\propto 1/E^{3.5}$ ) dominance over this region. Afterward, at medium energy regions (0.5–1.33 MeV), the decrements in MAC values are insignificant as the Compton scattering (CS) ( $\propto 1/E$ ) phenomenon dominates. As a conclusion, CoNi3 has superior effectiveness as a shielding material.

## Introduction

Nanotechnology develops a new branch for improving the properties of materials. In past days the investigation of nanoparticles has been performed by different researchers [1]. The growth of transition metal investigations such as tricobalt tetroxide and NiO which are interesting and favorable transition oxide due to their importance in many implementations like a store of data, gas detecting, catalysts, and electrochemical instruments [1–3]. Though improvement of technology has made human being life easier, this improvement has disadvantages like quick development of utilizing radioactive sources in our daily life including manufactures, medicinal diagnostic facilities, reactors, radiation waste store places, radiation study institutes, the nourishment sterilization, and medicinal therapy [4–6]. Nanocomposites materials have been studied by many researchers, Jabeen et al. were recorded that the (In<sub>2</sub>O<sub>3</sub>)<sub>30</sub>/(NiO)<sub>70</sub> nanocomposite sample is an appropriate nominee for electrode material in electrochemical application [7]. Gupta and Ahmed studied the structure, magnetic, and optical properties of Co<sub>3</sub>O<sub>4</sub>-

NiO nanocomposites samples [8]. The Electrochemical, mechanical and adhesive properties of surface-modified NiO-epoxy nanocomposite coatings on mild steel have been done by Xavier [9]. Alghazzawi et al. used the microwave hydrothermal method for the synthesis of rGO/NiO nanocomposite samples. They also recorded that the rGO/NiO nanocomposite is used as a sensitive nonenzymatic glucose sensor [10].

Due to the mischievous influences of ionizing radiation, the safeguard of humans against radiation has become a stringent problem. Thus, providing a suitable protective material is permanently very important in the radiation safety field [11]. Among all kinds of radiation, protection against gamma has become large attention because of their extremely penetration nature. Despite the high competence of traditional Pb and concrete in attenuate gamma, they have some disadvantages [12]. For example, it is hard to be confirmed of concretes efficiency in radiation shielding due to their moisture changing and non-homogeneity. Also, the poisonous nature of Pb has been performed in being deserted by numerous industries [13].

The protection strength of the shield depends on three parameters;

\* Corresponding author.

E-mail address: [drhafizzaid2020@gmail.com](mailto:drhafizzaid2020@gmail.com) (M.H.M. Zaid).

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radiation energy, the absorbent density, and Z in the shield [14,15]. Thus, there are two ways to progress the protection features of the shield: appropriate elements and the decrease of material pores [16]. Nanomaterials are excessively utilized in different applications. A nanoparticle is a very tiny particle generally with a size  $< 100$  nm, that could be utilized in materials. Nanomaterials are very attractive because of the very tiny size and high particular surface area and have a high possibility for beneficent material features like compressive strength and permeability [17]. In this regard, the nanoparticle performs some special tasks as a radiation shield; it could be reducing gamma. Besides, its properties can be easily changed by changing its composition. The study of gamma radiation shielding for NiO/ $x$ Co<sub>3</sub>O<sub>4</sub> nanocomposites prepared samples is novel because there is no such study in the literature. In addition, the novelty of this study is the use of the FLUKA Monte Carlo code to evaluate the mass attenuation coefficient for NiO/ $x$ Co<sub>3</sub>O<sub>4</sub> nanocomposites samples in the photon energy range from 0.1 to 1.33 MeV.

### Experimental techniques

The samples were prepared using a microwave oven (650 W) [18]. The starting materials are cobalt nitrate hexahydrate and nickel nitrate hexahydrate were used. The purity of these materials is equal to 97%. For pure Co<sub>3</sub>O<sub>4</sub> and, NiO, for example (Co<sub>3</sub>O<sub>4</sub> NPs), it was fabricated by adding twenty-five (ml) H<sub>2</sub>O solution of 0.2 mol. cobalt nitrate hexahydrate, with a 25 ml H<sub>2</sub>O solution containing 0.2 mol in a flask. The same procedures were used for the preparation NiO NPs using the starting material of Nickle nitrate hexahydrate. The solution was put in a microwave furnace operating at a maximum of 20 min.

The fine powder of Co<sub>3</sub>O<sub>4</sub> and NiO-NPs is collected for descriptions. For mixed nanocomposites of NiO/Co<sub>3</sub>O<sub>4</sub> were fabricated via the same process. Various contents of (30, 50, and 70 wt%) of cobalt nitrate hexahydrate are mixed with (70, 50 and 30 wt%) of nickel(II) nitrate hexahydrate with 0.2 mol. A fine powder of (NiO/Co<sub>3</sub>O<sub>4</sub>) nanocomposites is taken away. The samples (1- $x$ )NiO/ $x$ Co<sub>3</sub>O<sub>4</sub> with  $x = 30, 50$ , and 70 wt% are called CONi1, CONi2, and CONi3, respectively. This method for preparing nanoparticle is considered as a promising technique due to the easy and sheep aspects. Moreover, the smallest preparation time is one of the most features. The prepared samples are fine powder possessing uniform particle size. XRD measurements were carried via a Shimadzu XD-3A X-ray diffractometer CuK $\alpha$  radiations ( $\lambda = 0.15418$  nm).

FLUKA is a radiation transport code based on the Monte Carlo method. FLUKA code [19–21] was developed collaboratively by CERN and INFN, can be found elsewhere (Fig. 1) [22–24]. FLUKA code was utilized to evaluate the shielding features of three samples of (1- $x$ )NiO/ $x$ Co<sub>3</sub>O<sub>4</sub>, where ( $x = 0.3, 0.5$ , and  $0.7$ ) nanocomposites against  $\gamma$ -rays. In the present simulation, The BEAMPOS card was used to define the

position and direction of the radioactive source which is considered as a monoenergetic (0.2 cm dimension), a mono-directional beam of photons along plus z-axis with different energies at  $15\text{--}15 \times 10^3$  KeV. Then the angular and energy distribution of the source and the type of particle and its energy was known by the BEAM card. For photon transport at low energies, the energy cut-off is  $10^{-6}$  GeV by EMFCUT card. The photons were detecting via NaI (TI) ( $7.62 \times 7.62$  cm) scintillation detector. The detector was placed inside the lead cylinder collimator has a 120 and 2 mm outer and inner diameter, respectively with a 130 mm height. The NaI area was realized as the track-length fluence evaluator via the USRTRACK scorecard. In geometry, the sample was located between the source, and the detector surface is indicated in Fig. 1.

MATERIAL card describing the chosen sample contains the mixture name, weight fraction, specimen number, and density, etc. and it's used with wanted COMPOUND cards to determine the exact composition of the sample. Simulation processes run between  $10^6$  and  $20^6$  number of primary photons to obtain a statistical error  $< 1\%$ . The fluence of a photon in the volume of a detector was assessed with the USRBIN card. The sample was modeled as a cylinder, 40 mm diameter, with the different thicknesses from 2 to 5 mm. Glass samples were conducted with the Rectangular Parallelepiped body. RPP is characterized by 6 numbers  $X_{(\min \text{ and max})}$ ,  $Y_{(\min \text{ and max})}$ ,  $Z_{(\min \text{ and max})}$  and has edges parallel to the coordinate axes. In this simulation geometry,  $X_{(\min \text{ and max})}$ , and  $Y_{(\min \text{ and max})}$  were chosen as  $-50$  and  $+50$  mm, respectively. Photons passing through the sample have been detected in detector volume.

### Results and discussion

#### XRD measurements

Fig. 2 shows the XRD diffraction pattern of different compositions of (1- $x$ )NiO/ $x$ Co<sub>3</sub>O<sub>4</sub> NPs of (30, 50, and 70 wt%). As shown in Fig. 2, the 0.7NiO/0.3Co<sub>3</sub>O<sub>4</sub> NPs nanocomposites show eight peaks, which indicated to NiO NPs [25,26] with 3 additional three peaks at the angle of  $44^\circ$ ,  $52^\circ$ , and  $76^\circ$ . These three peaks are related to 30 wt% Co<sub>3</sub>O<sub>4</sub> NPs. With increasing Co<sub>3</sub>O<sub>4</sub> NPs content 0.5NiO/0.5Co<sub>3</sub>O<sub>4</sub> NPs, the intensity of these 3 peaks enhanced as illustrated in Fig. 2. For the 0.3NiO/0.7Co<sub>3</sub>O<sub>4</sub> NPs as shown in Fig. 2, these distinguish peaks are shifted to angles of  $32^\circ$ ,  $59^\circ$ , and  $65^\circ$ . This indicates a new phase of the p-p nanocomposite. The crystalline size of the NiO/Co<sub>3</sub>O<sub>4</sub> NPs with different concentrations of (30, 50, and 70 wt%) could be calculated using the following expression [27]:

$$D = \frac{0.94\lambda}{\beta \cos\theta} \quad (1)$$

where,  $\lambda$  and  $\beta$  are the wavelength and FWHM of the diffraction peak,

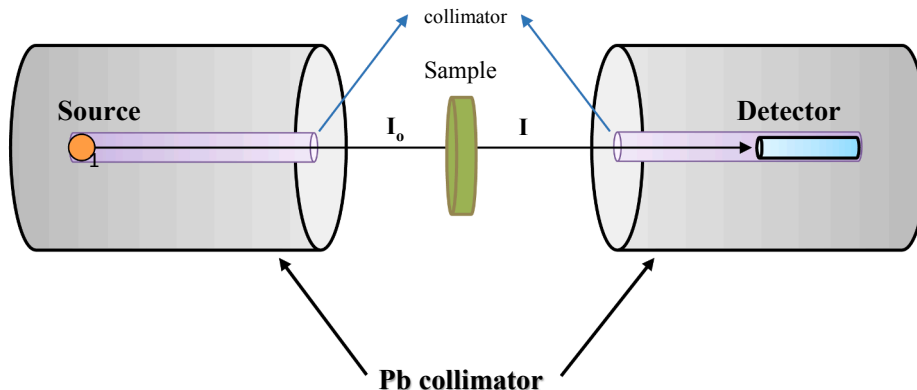


Fig. 1. FLUKA Monte Carlo simulation setup used for mass attenuation coefficients calculations of glasses.

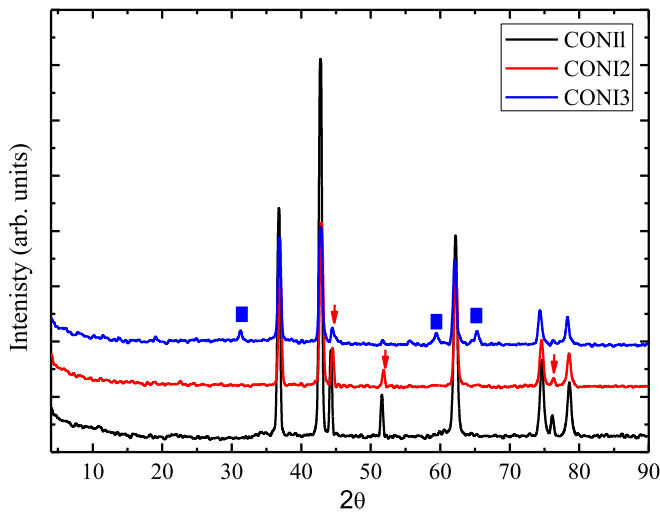


Fig. 2. XRD pattern of (100-x)NiO/xCo<sub>3</sub>O<sub>4</sub> composites.

respectively. The evaluated values of crystalline size are in the range of fourteen nm. The ionic radius of the host material of Ni<sup>2+</sup> is 69 pm [28]. On the other hand, the Co<sup>2+</sup> ionic radius is 65 pm [29]. Substitution alloys are expected because of that the host and foreign material are nearly similar.

#### Shielding parameters

If the radiation source is positioned between the counter detector and the absorber it decreases exponentially due to Beer-Lambert law and gamma rays that pass through the attenuator [30–32]:

$$I = I_0 e^{-\mu x} \quad (2)$$

where  $I_0$  is the intensity of non-attenuated and  $I$  is the intensity of attenuated gamma during the specimen. Also,  $\mu$  represents the linear attenuation coefficient and  $x$  is the specimen thickness. In the case of a compound, the mass attenuation coefficients ( $\mu_m$ ) are evaluated via the compound rule.

$$\mu_m = \sum_i w_i (MAC)_i \quad (2)$$

where  $w_i$  is the weight fraction of the  $i^{\text{th}}$  constitute elements. The desired sample thickness should reduce the absorbed gamma to one-half are called the half-value layer (HVL), and are acquired using the following equations [33–35]:

$$HVL = \frac{\ln(2)}{LAC} \quad (3)$$

Samples with a thickness of one mean free path (MFP) absorb 0.368 of the initial gamma intensity when it passes through the samples. It is calculated by [31,36,37]:

$$MFP = \frac{1}{LAC} \quad (4)$$

Gamma transmission ( $T$ ) of the studied samples was evaluated for 356, 662, 1173, and 1332 keV. The changes of evaluated  $T$  were presented against sample thickness ( $x$ ) in Fig. 3 for the CONI2 sample (an example). When  $x$  of the samples increases, the  $T$  values reduce quickly and increase as the photon energy increase. Fig. 4 shows the  $T$  values against sample thickness for all studied samples. As shown in this figure, the CONI3 sample has the lowest  $T$  among the studied samples. It is also evident from Figs. 3 and 4 the  $x$ -dependent vary of the  $T$ s moves away from linearity with increment energy. This indicates that for high energy the specimen thickness must be increased. For all CONI1, CONI2, and

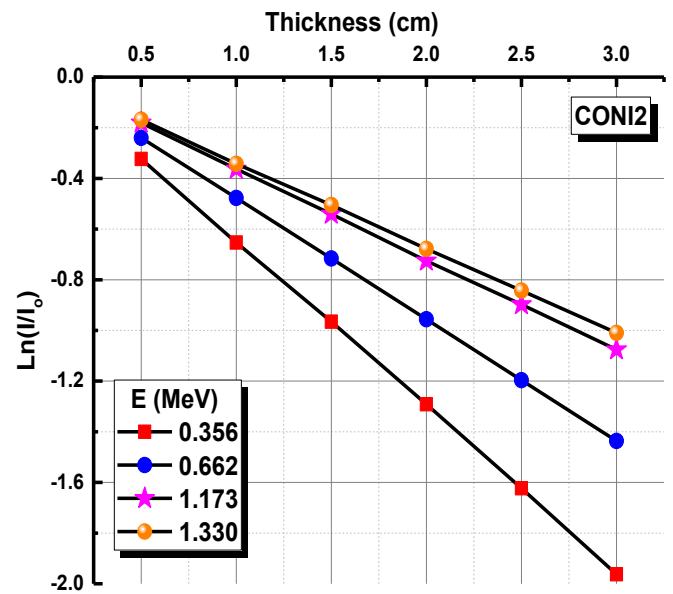


Fig. 3. Variation of transmission photon flux as a function of CONI2 sample thicknesses.

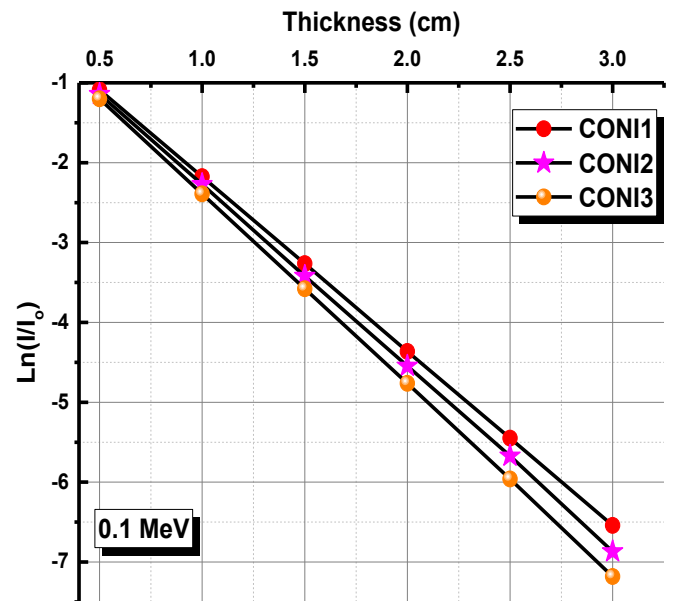


Fig. 4. Variation of transmission photon flux as a function of samples thicknesses at 0.1 MeV.

CONI3 samples Fig. 5 illustrates the changes in MAC, derived by applying FLUKA code at 0.1–1.33 MeV. It can be seen from Fig. 5 that with  $\gamma$ -ray energy increment from 0.1 to 1.33 MeV, from CONI1 to CONI3 samples, MAC values follow the same trend and improved noticeably with growing Co<sub>3</sub>O<sub>4</sub> compound. Sample CONI3 possesses comparatively greater MAC values owing to bigger wt. % of Co<sub>3</sub>O<sub>4</sub> in it, in all investigated glasses. For instance, at 0.1 MeV, 0.3472, 0.3567, 0.3673 (cm<sup>2</sup>/g) are the calculated MAC values for CONI1, CONI2, and CONI3 specimens, respectively. At low energies (0.1–0.5 MeV), for CONI1, CONI2, and CONI3 specimens, the noticed speedy reduction in MAC values indication that photoelectric effect (PEA) ( $\propto 1/E^{3.5}$ ) dominance over this region. Afterward, at medium energy regions (0.5–1.33 MeV), the decrements in MAC values are insignificant as the Compton scattering (CS) ( $\propto 1/E$ ) phenomenon dominates [38,39].

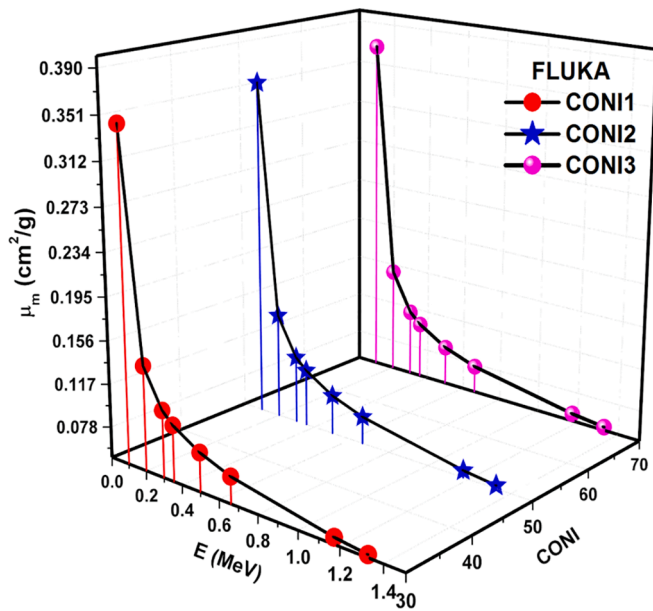


Fig. 5. Mass attenuation coefficient ( $\mu_m$ ) values for investigated samples against photon energy.

HVL, TVL, and MFP are indispensable parameters in assessing a material's gamma shielding competence, and commonly, the lower these values, the higher the shielding performance concerning sample thickness requirements. It is well known that lower HVL, TVL, and MFP refer to excellent protection capacity [20–24,33]. These factors with the same behavior of change against photon energy and sample compositions are given in Figs. 6–8, respectively. The calculated HVL, TVL, and MFP values change for CONI1, CONI2, and CONI3 samples within 356–1332 keV. Following Figs. 6–8, one can observe that for all investigated samples measured HVL, TVL, and MFP values show the same variance with photon energy. It is obvious that to decrease incident lower energy a very less glass thickness is enough. Then, with increasing photon energy, HVL, and MFP increase faster, approaching the corresponding largest values, indicating a bigger sample thickness requisite to reduce incoming gamma energy. As explained before, for  $\mu_m$  behaviors, The HVL, TVL, and MFP values depend on the incident photon energy corresponding to CS mechanisms dominates at medium energy.

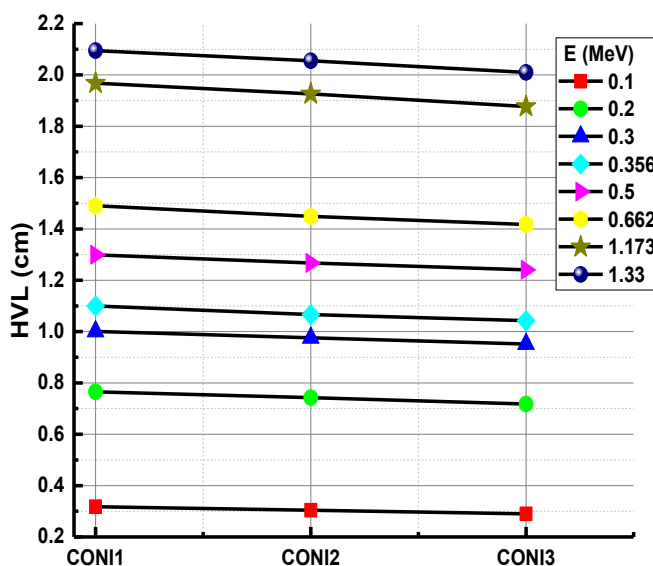


Fig. 6. Variation of HVL for investigated samples.

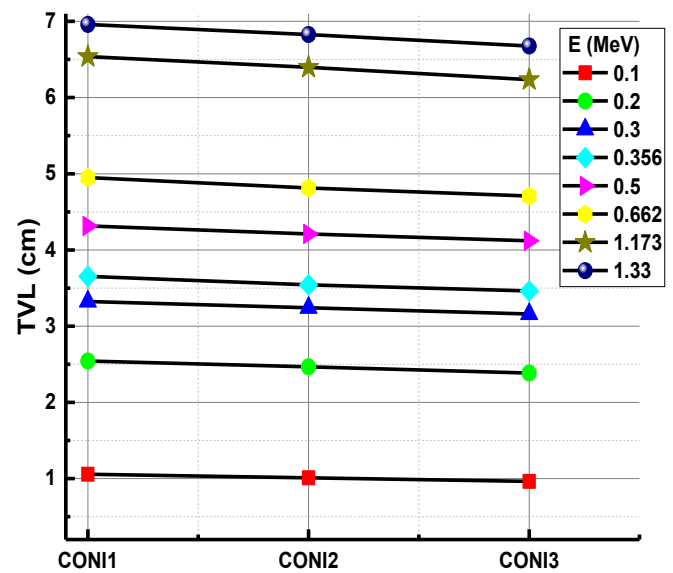


Fig. 7. Variation of TVL for investigated samples.

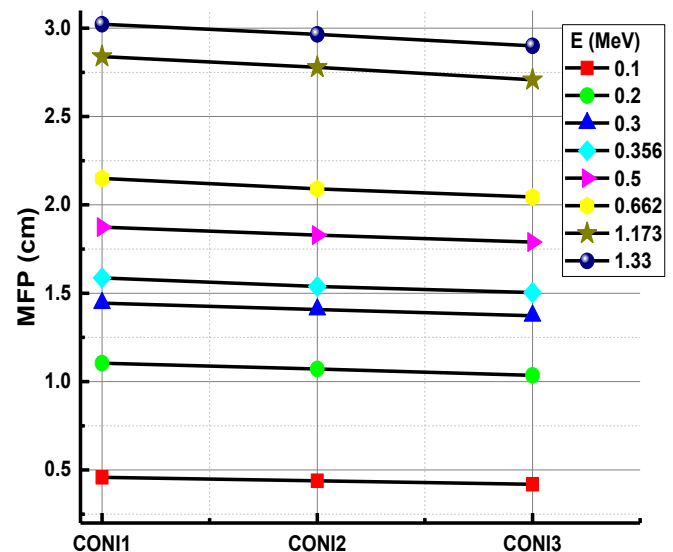


Fig. 8. Variation of MFP for investigated samples.

Furthermore, Figs. 6–8 show that HVL, TVL, and MFP values gradually decrease with increasing NiO concentration for CONI1, CONI2, and CONI3 samples. In all investigated samples, the relatively minimal thickness is required for sample CONI3 when compared to the rest of the samples to absorb photons as density improves for CONI1, CONI2, and CONI3 samples

## Conclusion

The (1-x)NiO/xCo<sub>3</sub>O<sub>4</sub> NPs of (30, 50 and 70 wt%). NPs were synthesized via the microwave irradiation method. These samples were checked using experimental XRD data. These obtained results confirmed the nature phase of the (1-x)NiO/xCo<sub>3</sub>O<sub>4</sub> nanocomposites. The sizes of these NPs were found to be in the range of 14 nm. Moreover, the addition of Co<sub>3</sub>O<sub>4</sub> improves the radiation shielding properties of (1-x)NiO/xCo<sub>3</sub>O<sub>4</sub> nanocomposites samples.



## CRediT authorship contribution statement

**Atif Mossad Ali:** Conceptualization, Methodology, Investigation, Writing - original draft. **Shams A.M. Issa:** Writing - original draft, Funding acquisition, Supervision. **Hesham M.H. Zakaly:** Writing - original draft, Supervision. **Mariia Pyshkina:** Conceptualization. **H.H. Somaily:** Investigation. **H. Algarni:** Conceptualization. **M. Rashad:** Conceptualization, Methodology. **M. Saif:** Conceptualization. **H.A.A. Sidek:** Conceptualization, Methodology. **K.A. Matori:** Conceptualization, Methodology. **M.H.M. Zaid:** Writing - original draft, Funding acquisition, Supervision.

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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